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REPUBLIEK VAN SUID-AFRIKA



Certificate

REPUBLIC OF SOUTH AFRICA

PATENT KANTOOR
DEPARTEMENT VAN HANDEL
EN NYWERHEID

PATENT OFFICE
DEPARTMENT OF TRADE AND
INDUSTRY

Hiermee word gesertifiseer dat
This is to certify that

REC'D 29 NOV 2004

WIPO PCT

the documents annexed hereto are true copies of:

Application forms P.1 and P.3, and the provisional specification of South African Patent Application No. 2003/8964 as originally filed in the Republic of South Africa on 18 November 2003 in the name of SASOL TECHNOLOGY (PROPRIETARY) LIMITED for an invention entitled: " PRODUCTION OF OXYGENATED PRODUCTS".

AND it is further certified that an application for amendment was filed at the South African Patent Office on 26 May 2004 to correct the names of two of the inventors as follows: WALCZUK, EDYTA to WALCZUK-GUSCIORA, EDYTA B AND KAMER, PAULUS C J to KAMER PAULUS CLEMENS JOZEF, and the amendment was allowed on 27 May 2004.

Geteken te
Signed at
PRETORIA

in die Republiek van Suid-Afrika, hierdie
in the Republic of South Africa, this

8

dag van
November 2004
day of

.....
Registrar of Patents

**PRIORITY
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REPUBLIC OF SOUTH AFRICA
PATENTS ACT, 1978
DECLARATION AND POWER OF ATTORNEY

(Section 30 - Regulation 8, 22(i)(o) and 33)

FORM P.3

PATENT APPLICATION NO		
21	01	2003/8964

A&A Ref: V16013

LODGING DATE	
22	18 November 2003

FULL NAME(S) OF APPLICANT(S)

71 SASOL TECHNOLOGY (PROPRIETARY) LIMITED

FULL NAME(S) OF INVENTOR(S)

72 VAN LEEUWEN, Petrus Wilhelmus Nicolaas Maria
WALCZUK-GUSCIORA, Edyta B
GRIMMER, Neil Eugene
KAMER, Paulus Clemens Jozef

EARLIEST PRIORITY CLAIMED	COUNTRY	NUMBER	DATE
	33 NIL	31 NIL	32 NIL

NOTE: The country must be indicated by its International Abbreviation - see schedule 4 of the Regulations

TITLE OF INVENTION

54 PRODUCTION OF OXYGENATED PRODUCTS

* I/We Morné Barradas

hereby declare that :-

~~I/we am/are the applicant(s) mentioned above;~~

*2. I/we have been authorized by the applicant(s) to make this declaration and have knowledge of the facts herein stated in the capacity of Authorized Signatory of the applicant(s);

**3. the inventor(s) of the abovementioned invention is/are the person(s) named above and the applicant(s) has/have acquired the right to apply by virtue of an assignment from the inventor(s);

4. to the best of my/our knowledge and belief, if a patent is granted on the application, there will be no lawful ground for the revocation of the patent;

~~5. this is a convention application and the earliest application from which priority is claimed as set out above is the first application in a convention country in respect of the invention claimed in any of the claims; and~~

the partners and qualified staff of the firm of ADAMS & ADAMS, patent attorneys, are authorised, jointly and severally, with powers of substitution and revocation, to represent the applicant(s) in this application and to be the address for service of the applicant(s) while the application is pending and after a patent has been granted on the application.

SIGNED THIS 17 DAY OF May 2004



Company Name: SASOL TECHNOLOGY (PROPRIETARY) LIMITED

Full Names:

Capacity: MORNE BARRADAS

IP LEGAL ADVISOR

(no legalization necessary)

*In the case of application in the name of a company, partnership or firm, give full names of signatory/signatories, delete paragraph 1, and enter capacity of each signatory in paragraph 2.

**If the applicant is a natural person, delete paragraph 2.

***If the right to apply is not by virtue of an assignment from the inventor(s), delete "an assignment from the inventor(s)" and give details of acquisition of right.

****For non-convention applications, delete paragraph 5.

EPUBLIC OF SOUTH AFRICA
TENTS ACT, 1978
PLICATION FOR A PATENT AND
CKNOWLEDGEMENT OF RECEIPT
ection 30(1) Regulation 22)

HE GRANT OF A PATENT IS HEREBY REQUESTED BY THE UNDERMENTIONED APPLICANT
N THE BASIS OF THE PRESENT APPLICATION FILED IN DUPLICATE

21 01 PATENT APPLICATION NO 2003 / 8964 A&A REF V16013 GSK

REPUBLIC OF SOUTH AFRICA
FORM P.1 REVENUE
(to be lodged in duplicate)

18.11.03

R 060.00

71 FULL NAME(S) OF APPLICANT(S)

SASOL TECHNOLOGY (PROPRIETARY) LIMITED

ADDRESS(ES) OF APPLICANT(S)

1 Sturdee Avenue, Rosebank, 2196 Johannesburg, Republic of South Africa

54 TITLE OF INVENTION

"PRODUCTION OF OXYGENATED PRODUCTS"

Only the items marked with an "X" in the blocks below are applicable.

THE APPLICANT CLAIMS PRIORITY AS SET OUT ON THE ACCOMPANYING FORM P.2. The earliest priority claimed is

Country:

No:

Date:

THE APPLICATION IS FOR A PATENT OF ADDITION TO PATENT APPLICATION NO 21 01

THIS APPLICATION IS A FRESH APPLICATION IN TERMS OF SECTION 37 AND BASED ON

APPLICATION NO 21 01

THIS APPLICATION IS ACCCOMPANIED BY:

A single copy of a provisional specification of 17 pages

Drawings of sheets

Publication particulars and abstract (Form P.8 in duplicate) (for complete only)

A copy of Figure of the drawings (if any) for the abstract (for complete only)

An assignment of invention

Certified priority document(s). (State quantity)

Translation of the priority document(s)

An assignment of priority rights

A copy of Form P.2 and the specification of RSA Patent Application No 21 01

Form P.2 in duplicate

A declaration and power of attorney on Form P.3

Request for ante-dating on Form P.4

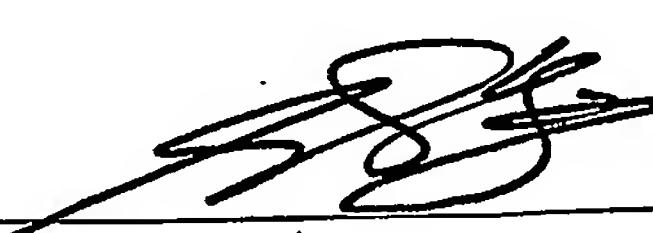
Request for classification on Form P.9

Request for delay of acceptance on Form P.4

Extra copy of informal drawings (for complete only)

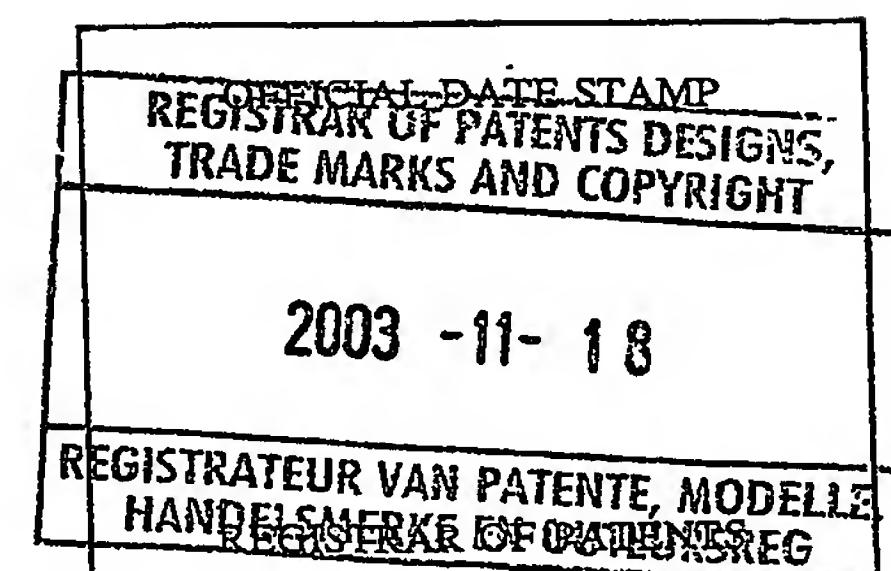
74 ADDRESS FOR SERVICE: Adams & Adams, Pretoria

Dated this 18 day of November 2003


ADAMS & ADAMS
APPLICANTS PATENT ATTORNEYS

The duplicate will be returned to the applicant's address for service as
proof of lodging but is not valid unless endorsed with official stamp

A&A P201



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PATENT ATTORNEYS
PRETORIA

REPUBLIC OF SOUTH AFRICA
Patents Act, 1978

PROVISIONAL SPECIFICATION
(Section 30 (1) - Regulation 27)

21 01 OFFICIAL APPLICATION NO

22 LODGING DATE

26 May 2004

71 FULL NAME(S) OF APPLICANT(S)

SASOL TECHNOLOGY (PROPRIETARY) LIMITED

72 FULL NAME(S) OF INVENTOR(S)

VAN LEEUWEN, PETRUS WILHELMUS NICOLAAS MARIA
WALCZUK-GUSCIORA, EDYTA B
GRIMMER, NEIL EUGENE
KAMER, PAULUS CLEMENS JOZEF

54 TITLE OF INVENTION

"PRODUCTION OF OXYGENATED PRODUCTS"

THIS INVENTION relates to the production of oxygenated products. It relates in particular to a process for producing oxygenated products from an olefinic 10 feedstock.

The production of aldehydes and alcohols is conveniently accomplished by employing a transition metal hydroformylation catalyst, in the presence of carbon monoxide and hydrogen, to convert an olefinic substrate or feedstock. The 15 addition of a phosphorus containing compound has been found to be highly beneficial in that higher product linearities can be obtained under less severe operating conditions. A broad range of olefins can be hydroformylated using such transition metals modified with phosphorus containing ligands. However, it has been found that the olefinic feedstocks must be substantially pure, ie free of 20 compounds such as dienes, ketones and alkynes. Such compounds are detrimental to the catalyst performance. These compounds may result in irreversible deactivation of the catalyst or have an incubatory effect from which the active hydroformylation catalyst may be recovered only if it is able to react with it. Invariably a dramatic drop, if not complete loss, of hydroformylation 25 activity occurs as the catalyst is tied up with these incubated species due to their low reactivity. In particular, such compounds react, in preference to α -olefins, with the transition metal, to form species which react very slowly and therefore act as sinks removing catalyst from the hydroformylation process. Thus, a feedstock containing such components can have a catastrophic effect on a 30 hydroformylation process where complete cessation of hydroformylation activity can result. These catalyst inhibitors must therefore be removed by means of

purification procedures which are not only costly, but may result in the decrease of reactable α -olefins in the feedstock.

Fischer-Tropsch derived olefinic feedstocks contain, in addition to α -olefins which 5 are the desired olefins for hydroformylation to aldehydes and alcohols, also small amounts of other compounds such as other olefinic compounds, ie hydrocarbons having at least one double bond, being linear, branched or aromatic and not being α -olefins; dienes, conjugated and non-conjugated, with a terminal olefinic functionality or not; alkynes; ketones; aldehydes; carboxylates; carboxylic acids 10 and the like. Other feed components may include, where chemically possible, those consisting of combinations of these functionalities or/and with an α -olefin. As indicated hereinbefore, such compounds have hitherto been undesired in hydroformylation feedstocks, for the reasons given hereinbefore, and are hereinafter also referred to as 'undesired components'.

15 By 'Fischer-Tropsch derived' in respect of the olefinic feedstock is meant that the feedstock has been obtained by the so-called Fischer-Tropsch process, ie obtained by reacting a synthesis gas comprising carbon monoxide and hydrogen in the presence of a suitable Fischer-Tropsch catalyst, normally a cobalt, iron, or 20 cobalt/iron Fischer-Tropsch catalyst, at elevated temperature in a suitable reactor, which is normally a fixed or slurry bed reactor, to obtain a range of products; these products must then be worked up to obtain a Fischer-Tropsch derived olefinic stream, typically a C_2 to C_{20} olefinic stream, which is suitable for use as a feedstock to a hydroformylation process. This feedstock is 25 characterized thereby that it has not been worked up sufficiently to remove all inhibitory compounds hereinbefore described.

It is thus an aim of this invention to provide, in a hydroformylation process, a transition metal-ligand catalyst system by means of which such a Fischer-Tropsch derived olefinic feedstock can be hydroformylated directly. Thus, the catalyst system must either be more resistant to the negative influences of some 30

of the undesired compounds in the feedstock or react more rapidly with them, than has hitherto been the case.

Thus, according to a first aspect of the invention, there is provided a process for
5 producing oxygenated products from an olefinic feedstock containing, in addition to at least one α -olefin, also at least one undesired component, which process includes reacting the feedstock, in a hydroformylation reaction stage, with carbon monoxide and hydrogen at an elevated reaction temperature and at a superatmospheric reactor pressure in the presence of a hydroformylation catalyst
10 system comprising a mixture, combination or complex of

- (i) a transition metal, T, where T is selected from the transition metals of Group VIII of the Periodic Table of Elements;
- (ii) carbon monoxide, CO;
- (iii) hydrogen, H₂;
- 15 (iv) a monodentate phosphorus ligand; and
- (v) a bidentate phosphorus ligand.

The olefinic feedstock may, at least in principal, be that obtained from any olefin oligomerization process, such as one of those known under the trade names
20 SHOP, Phillips and Ziegler; metathesis; dehydration of alcohols; or dimerization, provided it contains at least one undesired component as hereinbefore described. However, it is envisaged that the process of the invention will have particular application to Fischer-Tropsch derived olefinic feedstocks, as hereinbefore described.

25 More particularly, T may be Co, Rh, Ir, Rh or Pd; however, rhodium (Rh) is preferred. Examples of rhodium sources that can be used are Rh(acac)(CO)₂, Rh(acac)(CO)(TPP) (acac = acetylacetone; TPP = triphenylphosphine), [Rh(OAc)₂]₂, Rh₂O₃, Rh₄(CO)₁₂, Rh₆(CO)₁₆, Rh(CO)₂(dipivaloyl methanoate) or
30 Rh(NO₃)₃. Preferably, the rhodium is initially in the form of Rh(acac)(CO)₂ or Rh(acac)(CO)(TPP). The catalyst system may initially be prepared by dissolving

the metal source together with the ligands in a suitable solvent. This catalyst solution is introduced into a hydroformylation reactor, and upon heating thereof in the presence of synthesis gas comprising CO and H₂, results in the formation of an active hydroformylation catalyst system.

5

The concentration of rhodium in the catalyst solution in the hydroformylation reactor may be from 10 to 1000 ppm, more preferably from 50 and 500 ppm, and most preferably from 50 and 300 ppm.

10 Ideally the ligands should be used in an excess concentration to the transition metal. The monodentate ligand can be used in a molar excess of from 5 to 2000, more preferably from 10 to 1000, and most preferably from 20 to 1000, to rhodium. The bidentate phosphorus ligand will, in many circumstances, preferentially bind to the transition metal, displacing the monodentate ligand. It
15 can therefore be employed at lower ligand to transition metal ratios such as 0.2:1 – 100:1, and more preferably from 0.5:1 – 50:1. Of course, in each catalyst system an ideal monodentate:bidentate:transition metal ratio would have to be determined depending not only on the properties of the ligands employed, but also the product specification desired as well as the composition of the feedstock.

20

The monodentate phosphorus ligand may be a monodentate phosphine or phosphite ligand. In one embodiment of the invention, the monodentate phosphorus ligand may be that of Formula (1), and which is hereinafter also referred to as Ligand 1a ('L1a'):

25 $P(R^a)(R^a)(R^a)$ (1)
where all R^a are the same or dissimilar, and are each a branched or straight chair alkyl or aryl radical. Preferably, however, each R^a is an aryl group, and all R^a are the same. Most preferably, each R^a may be phenyl so that L1a is then triphenylphosphine ('TPP').

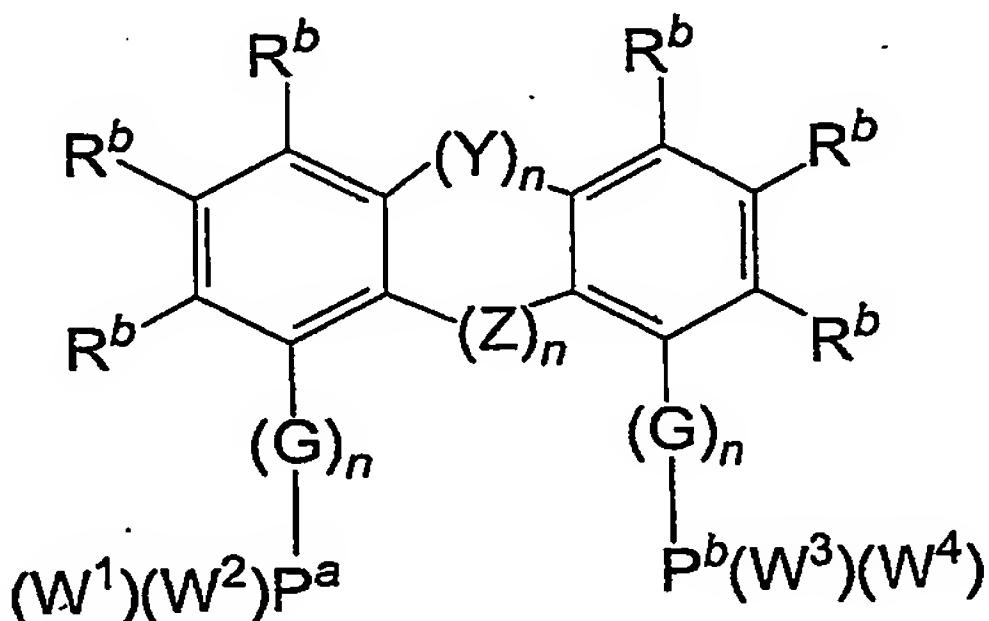
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However, in another embodiment of the invention, the monodentate ligand may be that of Formula (2), and which is hereinafter also referred to as Ligand 1b ('L1b'):



5 where R^a is as hereinbefore defined. Preferably, however, each R^a in Formula (2) is an aryl group, and all R^a are the same. Most preferably, each R^a may be a substituted phenyl ring. Thus, L1b may for example be tris(2,4-ditertiary butylphenyl) phosphite or tris(2-tertiary butylphenyl) phosphite.

10 The bidentate phosphorus ligand (which is hereinafter also referred to as L2) may, in one embodiment of the invention, be that of Formula (3):



20 wherein

(i) all R^b are the same or dissimilar, and are each H, alkyl, alkoxy, cycloalkyl, cycloalkoxy, heterocycloalkyl, aryl, heteroaryl, aryloxy, polyether, cyano, nitro, halogen, trifluoromethyl, $-C(O)R^c$, $-(R^d)C(O)R^c$, $-CHO$, $(R^d)CHO$, $-COOR^c$, $-(R^d)COOR^c$, $-COO^-M^+$, $-(R^d)COO^-M^+$, $-SO_3R^c$, $-(R^d)SO_3R^c$, $-SO_3^-M^+$, $-(R^d)SO_3^-M^+$, $-SR^c$, $-(R^d)SR^c$, $-SOR^c$, $-R^d(SOR^c)$, $-NR^c$, $-(R^d)NR^c$, $-N^+(R^c)(R^c)(X^-)$ or $-(R^d)N^+(R^c)(R^c)(X^-)$, wherein

25 (a) R^c and R^d are the same or different, and are each H, or a branched or straight chain alkyl, alkoxy, cycloalkyl, polyether, cycloalkoxy, heterocycloalkyl, aryl, heteroaryl or aryloxy radical;

30

(b) M^+ is a cation; and

(c) X^- is an anion;

5 (ii) Y and Z are independent bridges, are the same or different, and are each selected from the radicals, $-O-$, $-N(R^c)-$, $-N^+(R^c)(R^c)(X^-)-$, $-N(C(O)R^c)-$, $-C(R^c)(R^c)-$, $-C(C(R^c)(R^c))-$, $-C(O)-$, $-S-$, $-Si(R^c)(R^c)-$, $-Si(OR^c)(OR^c)-$, $-P(R^c)-$ or $-P(OR^c)-$, where R^c and X^- are as hereinbefore defined;

10 (iii) n (in $(Y)_n$ and $(Z)_n$) is, in each case, 0 or 1, with the proviso that n cannot be 0 for both Y and Z;

(iv) W^1 , W^2 , W^3 and W^4 are the same or different, and are each an alkyl (branched or straight chain), alkoxy, cycloalkyl, cycloalkoxy, heterocycloalkyl, aryl, heteroaryl, aryloxy or trifluoromethyl radical;

15 (v) a, b, in P^a and P^b , are used merely to identify the P atoms;

(vi) each G is an independent linker radical, are the same or different, and is selected from $-O-$, $-N(R^f)-$, $-N^+(R^f)(R^f)(X^-)-$, $-C(R^f)(R^f)-$, $-S-$, $-Si(R^f)(R^f)-$, $-C(F_2)-$ or $-C(R^f)(F)-$, wherein

20 (a) R^f is H, or a branched or straight chain alkyl, alkoxy, cycloalkyl, polyether, cycloalkoxy, heterocycloalkyl, aryl, heteroaryl or aryloxy radical, and with the proviso that when the radical contains more than one R^f , all R^f are the same or different;

(b) X^- is as hereinbefore defined; and

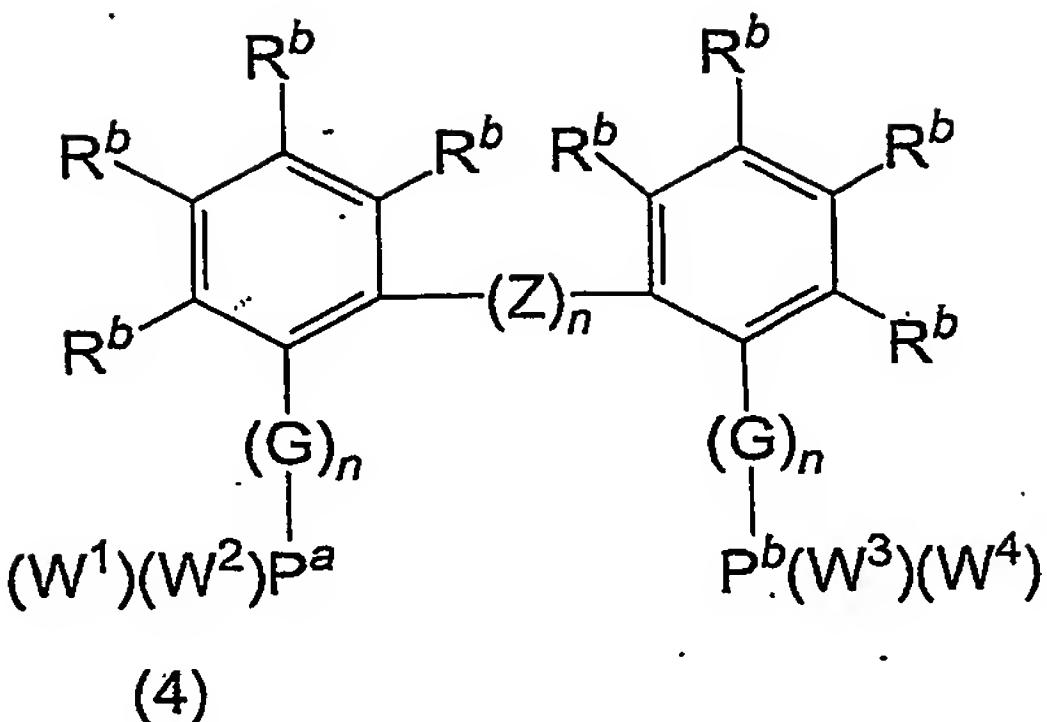
25 (vii) n (in each $(G)_n$) is 0 or 1.

M^+ may be an ion of an alkali or alkali earth metal, such as sodium, potassium or barium, or it may be ammonium or a quaternary ammonium ion.

25 X^- may be an organic acid, phosphate or sulphate group, for example $-CO_2^-$, $-PO_3^{2-}$ or $-SO_3^-$.

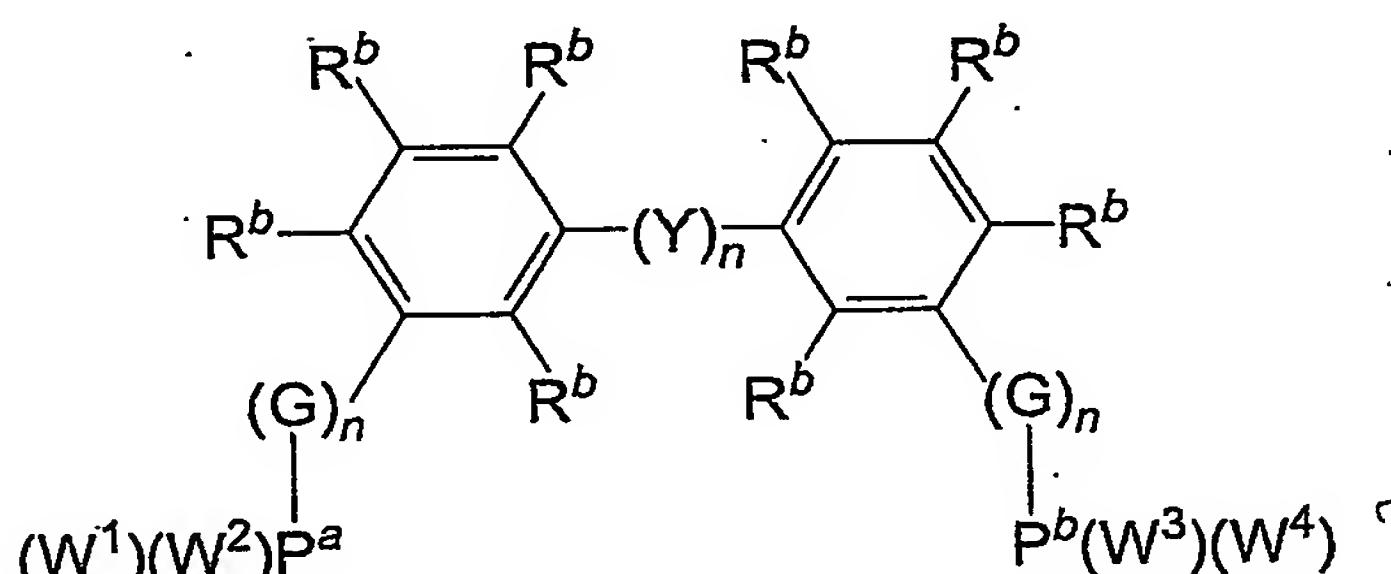
30 When $n = 0$, in $(Y)_n$, then the independent Y bridge is naturally absent. L2 will then be in accordance with Formula (4):

5



10 When $n = 0$, in $(Z)n$, then the independent Z bridge is naturally absent. L2 will then be in accordance with Formula (5):

15

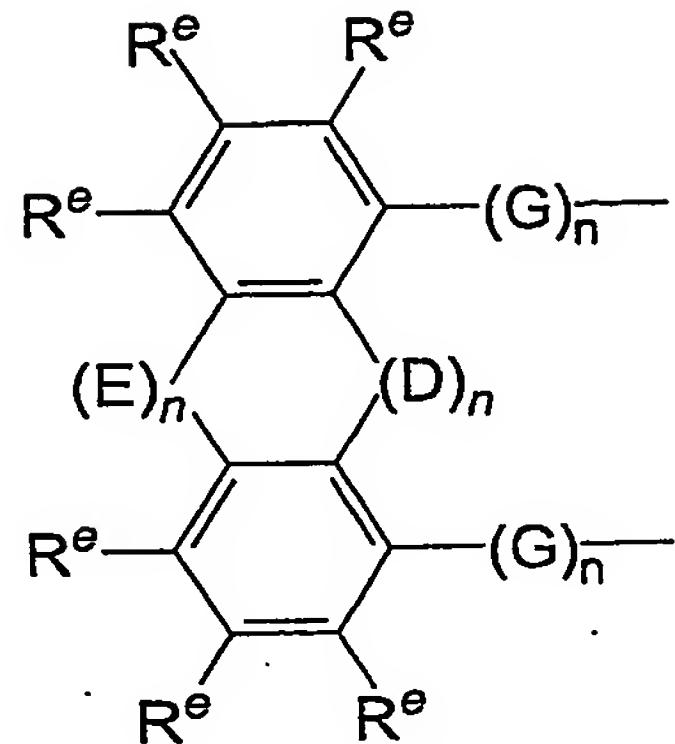


20

In L2 in accordance with Formulae (4) and (5), R^b , W^1 , W^2 , W^3 , W^4 , Y , Z and G are thus as hereinbefore defined.

25 W^1 , W^2 , W^3 and W^4 may, in particular, each be an alkyl, aryl or aryloxy radical; however, aryl and aryloxy radicals are preferred. Most preferred are aryl or aryloxy radicals represented by Formula (6); however, the structure of Formula (6) does not represent a bridging unit connecting P^a to P^b – for P^a , W^1 and W^2 represent radicals connected through their respective G linkers, and for P^b , W^3 and W^4 represent radicals connected through their respective G linkers; however, 30 for W^1 and W^2 on the one hand, and W^3 and W^4 on the other hand, G may be the same or different to that in L2 of Formula (3).

5



10

(6)

wherein

(i) all R^e are the same or different, and are each H, alkyl, alkoxy, cycloalkyl, cycloalkoxy, heterocycloalkyl, aryl, heteroaryl, aryloxy, polyether, cyano, nitro, halogen, trifluoromethyl, $-C(O)R^c$, $-(R^d)C(O)R^c$, $-CHO$, $(R^d)CHO$, $-COOR^c$, $-(R^d)COOR^c$, $-COO^-M^+$, $-(R^d)COO^-M^+$, $-SO_3R^c$, $-(R^d)SO_3R^c$, $-SO_3^-M^+$, $-(R^d)SO_3^-M^+$, $-SR^c$, $-(R^d)SR^c$, $-SOR^c$, $-R^d(SOR^c)$, $-NR^c$, $-(R^d)NR^c$, $-N^+(R^c)(R^c)(X^-)$ or $-(R^d)N^+(R^c)(R^c)(X^-)$, wherein R^c , R^d , X^- and M^+ are as hereinbefore defined;

15 (ii) G and n (in $(G)n$) are as hereinbefore defined;

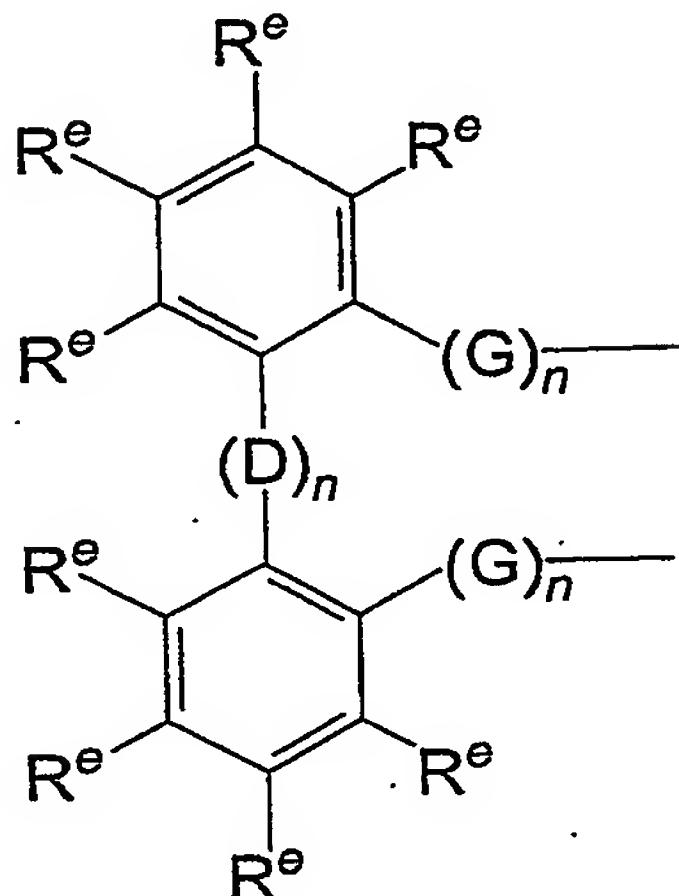
(iii) D and E are each an independent bridge, are the same or different, and are each selected from the radical, $-O-$, $-N(R^c)-$, $-N^+(R^c)(R^c)(X^-)$, $-N(C(O)R^c)-$, $-N(SiR_2^c)-$, $-C(R^c)(R^c)-$, $-C(C(R^c)(R^c))-$; $-C(O)-$, $-S-$, $-Si(R^c)(R^c)-$, $-Si(OR^c)(OR^c)-$, $-P(R^c)-$ or $-P(OR^c)-$, wherein R^c and X^- are as hereinbefore defined;

20 (iv) n (in each of $(D)n$ and $(E)n$) is 0 or 1.

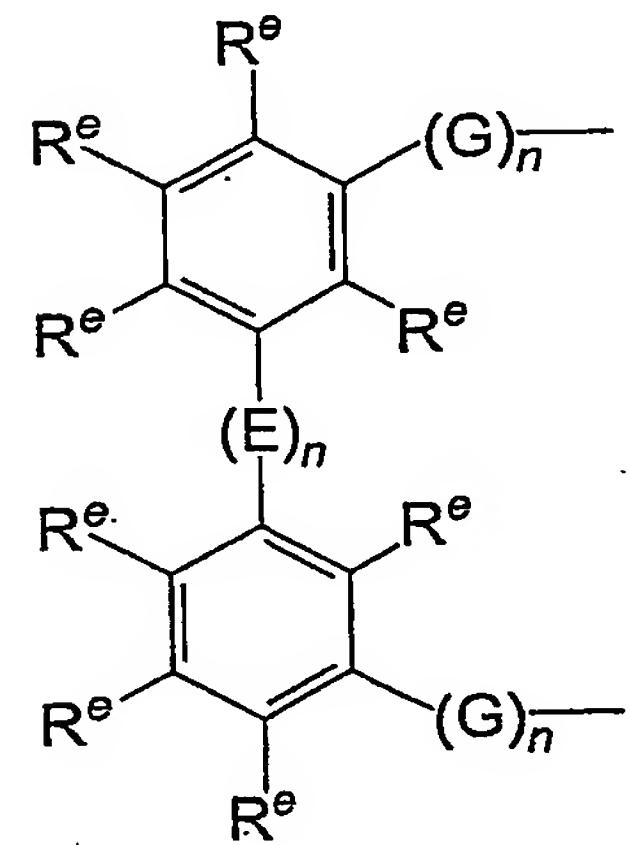
When D and/or E is present, W^1 and W^2 represent one diradical connected to the P atom. The same applies for W^3 and W^4 .

30

When $n = 0$, in $(E)_n$, then the independent E bridge is naturally absent. The structure of Formula (6) will then have the structure in accordance with Formula (7):

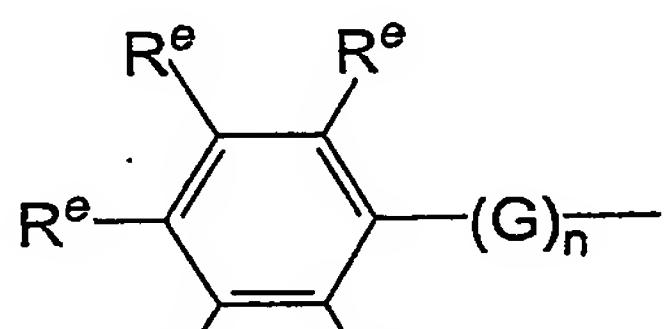


When $n = 0$, in $(D)_n$, then the independent D bridge is naturally absent. The structure of Formula (6) will then have the structure in accordance with Formula (8):

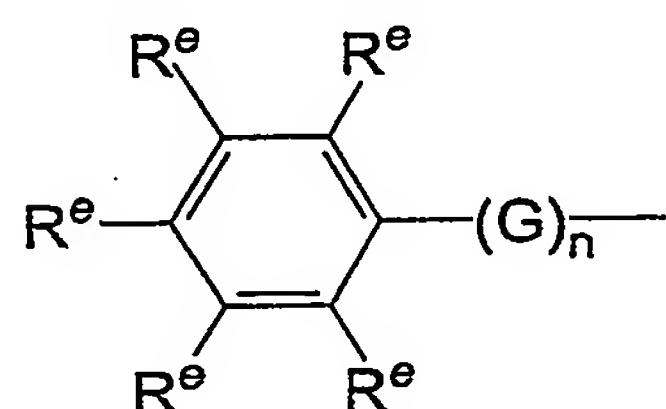


When $n = 0$, in both $(D)_n$ and $(E)_n$, then both the independent bridges D and E are naturally absent. The structure of Formula (6) will then have the structure in accordance with Formula (9) i.e. two separate, unbridged radicals:

5



10



(9)

15

In Formulae (7), (8) and (9), R^e , D, E and G are as hereinbefore defined.

When $n = 0$, in respect of $(G)_n$, then a direct connection exists between the P atom and the phenyl ring in the structures of Formulae (3), (6), (7), (8) and (9). In

20 such case, a phosphine substructure may be formed.

However, in another embodiment of the invention, L2 may be that of Formula (10):

25



(10)

where

- (i) P, G, W^1 , W^2 , W^3 and W^4 are as hereinbefore defined;
- (ii) A is a bridging unit and is selected from one of the following diradicals: –
 $(CR^b_2)_n-$, $-(CR^b)_n-$, $-(CR^bCR^b)_n-$, $-[C(O)]_n-$, $-[C(O)C(R^b)_2]_n-$, $-(NR^b)_n-$, –
 $S-$, $-(SiR^b_2)_n-$, $-(SiOR^b_2)_n-$, with

5

(a) any alkyl radical having $n = 1$ to 5 and being cyclic, straight or branched or straight;

(b) R^b being as hereinbefore defined; or

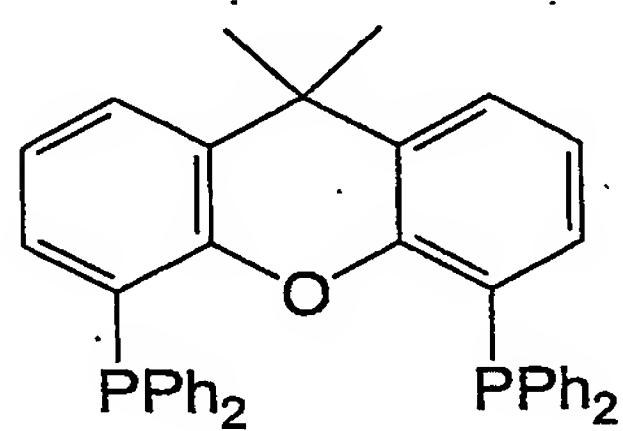
(iii) A is a bridging unit and is '-Ar-', which is an aryl or heteroaryl group of between 4 and 18 carbon atoms.

When, in Formula (10), $n = 0$ (in respect of $(G)n$), then a direct connection exists between a phosphorus atom and the bridging unit A in Formula (10). In such case, a phosphine substructure may be formed.

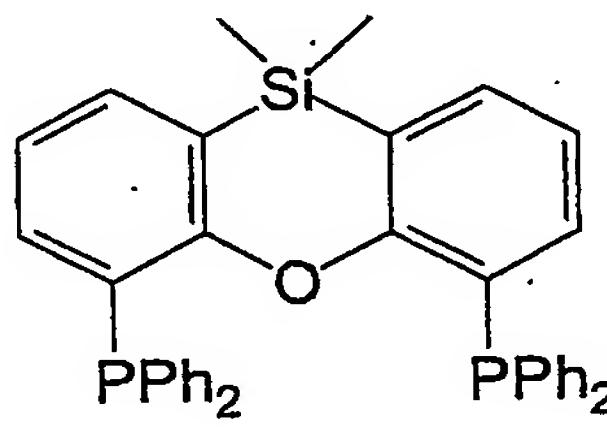
10

In Formulae (4), (5), (6), (7) and (8), the independent bridges X, Y, D and E may represent a direct linkage between the phenyl rings without an intervening group or atom.

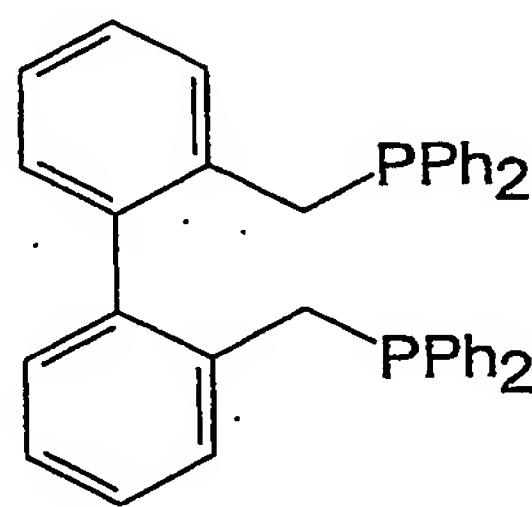
15 Ideally, the bidentate ligand, L₂, should have a wide bite angle, with those belonging to the xantphos family of ligands, and variations thereupon, being preferred examples. Examples of such preferred ligands L₂ are given in formulae (11) to (18) below:



(11)



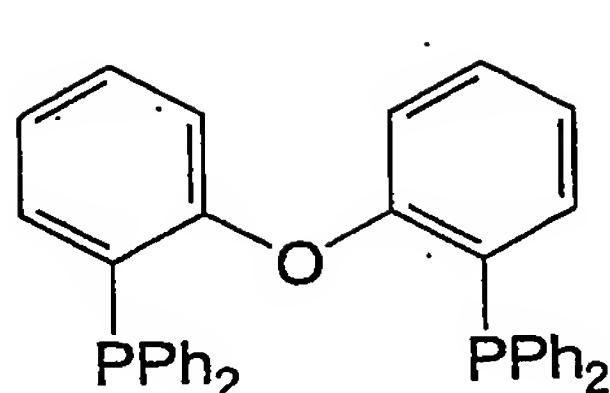
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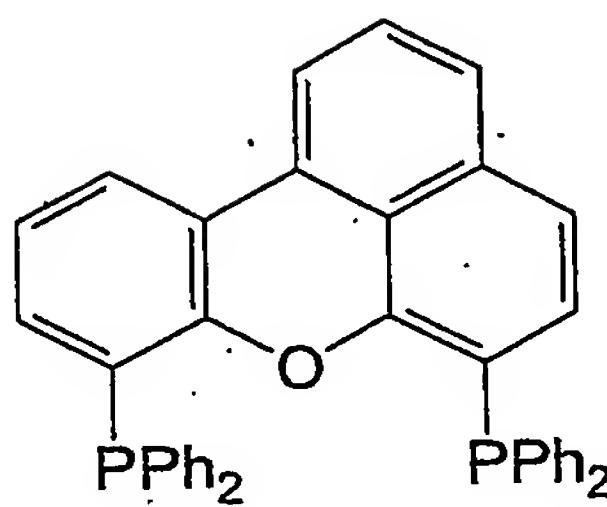
(13)

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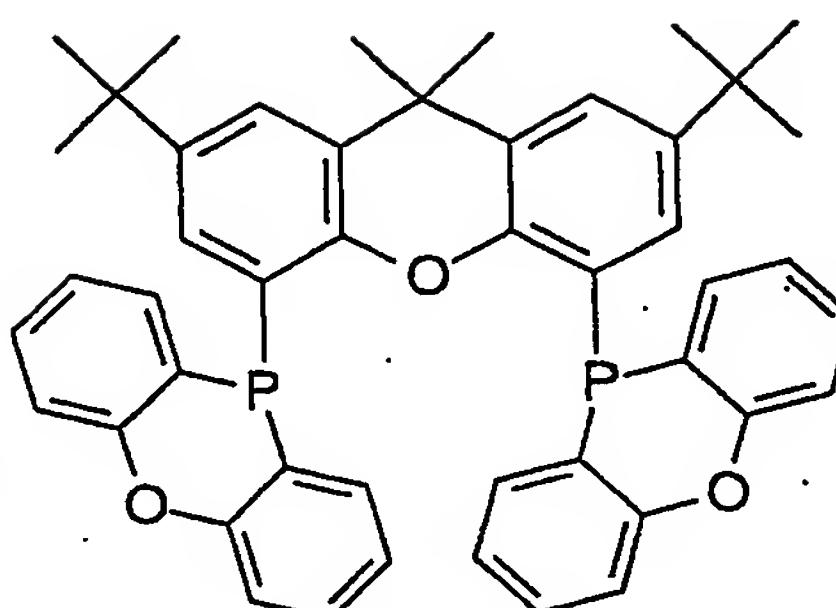
13



(14)



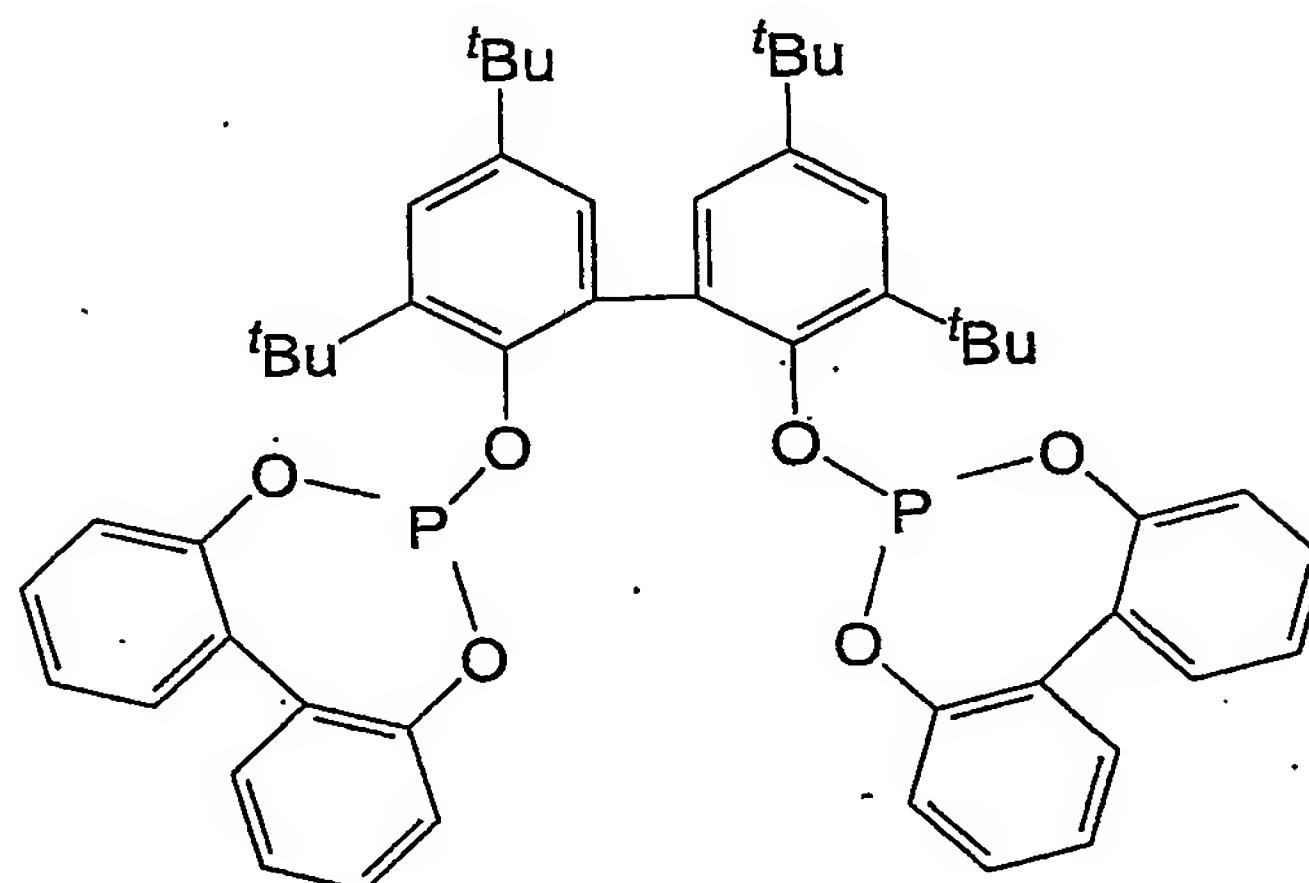
(15)



(16)



(17)



(18)

5

(where $\text{Ph} = \text{C}_6\text{H}_5$ in the above examples)

The temperature in the hydroformylation reactor may be from 50°C to 150 °C, more preferably from 70°C to 120 °C.

10

The synthesis gas pressure under which the hydroformylation reaction is performed may be from 1 to 100 bar, but more preferably from 5 to 40 bar, and most preferably from 10 to 30 bar. The $\text{H}_2:\text{CO}$ ratio may be from 1:10 to 100:1, but most preferably from 1:1 and 5:1.

15

According to a second aspect of the invention, there is provided a process for producing oxygenated products from an olefinic feedstock containing, in addition to at least one α -olefin, also at least one undesired component, which process includes reacting the feedstock, in a hydroformylation reaction stage, with carbon monoxide and hydrogen at an elevated reaction temperature and at a superatmospheric reactor pressure in the presence of a hydroformylation catalyst system comprising a mixture, combination or complex of

5 (i) a transition metal, T, where T is selected from the transition metals of Group VIII of the Periodic Table of Elements;

10 (ii) carbon monoxide, CO;

(iii) hydrogen, H₂;

(iv) a monodentate phosphorus ligand,

with the molar ratio of the monodentate phosphorus ligand to the transition metal T being at least 5:1.

15 Preferably, the molar ratio of monodentate phosphorus ligand to the transition metal T is from 5:1 to 2000:1, more preferably from 20:1 to 1000:1, and still more preferably from 50:1 to 1000:1.

20 As regards all other features, the process of the second aspect of the invention may be similar to that of the first aspect of the invention.

25 The invention will now be described in more detail with reference to the following non-limiting examples:

In all the Examples, autoclave experiments were performed in Parr autoclaves. The catalyst precursors were dissolved in toluene in a Schlenk tube under an argon atmosphere. This solution was then transferred via cannula to the autoclave which had been purged of air with argon. The reactor was sealed, flushed twice with synthesis gas and then pressurised with synthesis gas. The reactor contents were then heated and upon reaching reaction temperature the

substrate, ie the olefinic feedstock, was charged into the reactor, via a sample bomb, using synthesis gas overpressure. The progress of the reaction was monitored either by means of a mass-flow meter or the drop in pressure of a ballast vessel. Unless stated otherwise a 1:1 H₂:CO synthesis gas mixture was 5 employed in the experiments. All ratios or proportions are given on a molar basis.

EXAMPLE 1

Example 1a

10 Rh(acac)(CO)₂ (9.6 × 10⁻⁵ mol) and TPP (Rh:TPP = 1:170) were dissolved in 50 mL toluene, which was then transferred to a 100 mL reactor. 1-Octene (10 mL) spiked with methyl vinyl ketone (100 mol eq. to Rh) was injected into the reactor once reaction temperature had been reached. The reaction was performed at 15 bar pressure and 100 °C.

15 The time taken to reach 50 % olefin conversion was 1hr45min.

Example 1b

20 The same experimental procedure as described Comparative Example 1a was followed with the difference that 4,5-bis(diphenylphosphino)-9,9-dimethylxanthene (hereinafter referred to as xantphos) was added as a second ligand (Rh:TPP:Xantphos = 1:170:5).

The time taken to reach 50 % olefin conversion was 1 hr.

Example 1c

25 The same procedure as described for Comparative Example 1b was followed with the difference that the Rh:TPP:Xantphos ratio was changed to 1:170:3.

The time taken to reach 50 % olefin conversion was 1 hr.

Example 1d

The same procedure as described for Comparative Example 1b was followed with the difference that the Rh:TPP:Xantphos ratio was changed to 1:170:1.

The time taken to reach 50 % olefin conversion was 1hr30min.

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Example 1e

The same procedure as described for Comparative Example 1b was followed with the difference that the Rh:TPP:Xantphos ratio was changed to 1:90:5.

The time taken to reach 50 % olefin conversion was 35 min.

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EXAMPLE 2Example 2a

Rh(acac)(CO)₂ (9.0 $\times 10^{-5}$ mol) and TPP (Rh:TPP = 1:90) were dissolved in 130 mL toluene, which was then transferred to a 300 mL reactor. 1-Octene (20 mL) was charged into the reactor once reaction temperature had been reached. The reaction was performed at 90 °C and 20 bar synthesis gas pressure.

The time taken to reach 50 % olefin conversion was 13 min.

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Example 2b

The same experimental procedure as described in Comparative Example 2a was followed with the difference that the Rh:TPP was increased to 1:170.

The time taken to reach 50 % olefin conversion was 20 min

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Example 2c

The same experimental procedure as described in Comparative Example 2a was followed with the difference that the 1-octene feed was spiked with methyl vinyl ketone (100 mol eq. to Rh).

The time taken to reach 50 % olefin conversion was 3hr35min.

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Example 2d

The same procedures as for Comparative Example 2b with the difference that the 1-octene feed was spiked with methyl vinyl ketone (100 mol eq to Rh).

The time taken to reach 50 % olefin conversion was 2h50min.

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The Applicant has thus unexpectedly found that by using either a catalyst system comprising a Group VIII transition metal together with a monodentate phosphorus ligand/bidentate phosphorus ligand combination or a catalyst system comprising a Group VIII transition metal together with a monodentate phosphorus ligand, 10 with the ligand being used in a molar excess of at least 5:1 relative to the transition metal, in a hydroformylation process, an olefinic feedstock comprising at least one α -olefin and at least one undesired compound can be accommodated in the process. Thus, such an olefinic feedstock can then be treated in the hydroformylation process without unacceptable deactivation and/or 15 loss of activity of the catalyst occurring.

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DATED THIS 18TH DAY OF NOVEMBER 2003.



ADAMS & ADAMS
APPLICANTS PATENT ATTORNEYS

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